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Author: David Vaniman				
<p>Environmental Restoration Project Standard Operating Procedure</p> <p>for:</p> <h1>Operating the ADEM Scanning Electron Microscope</h1>				
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Revision Log

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Operating the ADEM Scanning Electron Microscope

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Operating the ADEM Scanning Electron Microscope

1.0 PURPOSE

This procedure provides instructions for the operation of the Automated Digital Electron Microscope (ADEM) scanning electron microscope (SEM).

2.0 SCOPE

This SOP is a mandatory document and shall be implemented by all ER Project participants when performing scanning electron microscopy for the ER Project

3.0 TRAINING

- 3.1 All users of this SOP are trained by reading the procedure. The **users** shall ensure the training is documented in accordance with QP-2.2, and is entered appropriately in the ER Project Training Database located at <http://erinternal.lanl.gov/Training/Training.asp>.
- 3.2 The **Geology Task Leader** will monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with QP-2.2.

4.0 DEFINITIONS

- 4.1 ADEM—Automated Digital Electron Microscope.
- 4.2 Machine Custodian—The Machine Custodian is responsible for ADEM SEM maintenance. This includes calibration of magnification and Energy Dispersive System (EDS) gain, EDS resolution check, software and electronically-stored system backups, and all instruction and training of Procedure Users. The Machine Custodian may delegate responsibilities for the ADEM SEM System including maintenance, calibrations, resolution check, and instruction to Procedure Users.

5.0 BACKGROUND AND PRECAUTIONS

- 5.1 The ADEM scanning electron microscope includes interlocked safety systems that prevent damage to either operator or instrument.

6.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

- 6.1 Geology Task Leader

6.2 Quality Program Project Leader

6.3 Users

6.5 ER Project Participants

7.0 EQUIPMENT AND SOFTWARE

The ADEM SEM (purchase date February 1989) is a fully integrated computer controlled SEM, EDS, and image analysis system. The ADEM Software and OS9, the operating system software, are commercially acquired software packages provided with the system.

8.0 PROCEDURE

Note: Subcontractors performing work under the ER Project's quality program may follow this standard operating procedure (SOP) for operating the ADEM scanning electron microscope or may use their own procedure(s) for comparable equipment as long as the substitute meets the requirements prescribed by the ER Project Quality Management Plan, and is approved by the ER Project's Quality Program Project Leader (QPPL) before the commencement of the activitie(s).

Note: ER Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/home_links/Library_proc.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

Note: Deviations from SOPs are made in accordance with QP-4.2, Standard Operating Procedure Development and documented in accordance with QP-5.7, Notebook Documentation for Environmental Restoration Technical Activities.

The ADEM SEM is capable of analytical work in the following general areas: elemental qualitative and semi-quantitative standardless analysis, semi-quantitative elemental analysis using standards, image analysis, particle analysis, elemental mapping, elemental line profiles, and composite analysis of X-Ray maps and images. Procedure Users may consult with the Machine Custodian for help in for determining what procedures are appropriate for their work.

The ADEM SEM is set up and operated according to instructions contained in the ADEM Operators Manuals. The manuals are stored in the laboratory near the instrument.

8.1 Sample Preparation - Most samples for SEM examination require a conductive coating to reduce surface charging. Coating procedures are detailed in the following manuals:

“Instruction Manual”, Ladd Vacuum Evaporator, cat. no. 3000, 27 pgs., Burlington, Vermont

“Hummer II Operation and Maintenance Manual”, Technics, 12 pgs. Alexandria, Virginia.

For details concerning selection of coating medium, operators may consult with the Machine Custodian.

- 8.2 Use of Standards - Standards traceable to National Institute of Standards and Technology (NIST, formerly National Bureau of Standards, NBS) or well characterized materials published in credible technical journals and widely used by microanalysts may be used for calibration of semi-quantitative X-Ray analysis routines (e.g. standards from the Smithsonian evaluated for homogeneity and published in GeoStandards Newsletter, Jarosewich, et.al., 1980). Principle Investigators and Procedure Users may use standards from other sources for specific applications but must document the basis for usage of these standards in their notebooks.
- 8.3 ADEM Operation - The ADEM SEM is set up and operated according to instructions contained in the ADEM Reference Manual. The manuals are stored in the laboratory near the instrument.
- 8.4 Data Acquisition and Reduction - Data may consist of either image information or elemental information. Image information may be photographed and elemental information may be printed on paper by following the instructions in the operator’s manuals. Each photograph or paper hardcopy will be labeled with the sample identification number and keyed the sample number in the User’s notebook. Image and elemental information may be stored on magnetic media for retrieval at a later date. Data acquisition and reduction software are commercial software provided with the ADEM SEM and are described in the Operator manuals.
- 8.5 Sample Control – Sample identification will be based on the unique identifier marked on the sample. This will typically be written on the sample mounting stub with an indelible marking pen.
- 8.6 Potential Sources of Error and Uncertainty - Potential sources of error or uncertainty will be indicated by the Procedure Users inability to obtain a quality image or to generate an acceptable semi-quantitative analysis.
- 8.7 Equipment Malfunctions - Malfunction of the ADEM SEM System is readily detectable by the Machine Custodian during operation of the instrument. If the Procedure User has concerns regarding possible equipment malfunction during operation, he/she should consult with the Machine Custodian.
- 8.8 Safety Considerations - Normal operating conditions as performed by trained Procedure Users present no safety hazards.

- 8.9 Environmental Conditions - Normal interior building temperature and humidity are acceptable for the operation of the ADEM SEM. Compressed air in the range of 50 to 80 psi is supplied by the building utilities. Cooling water for the ADEM SEM diffusion pump is supplied by a chilled water system maintained in the range of 55 to 65 degrees Fahrenheit. Ambient air temperature for the ADEM SEM should range between 60 and 80 degrees Fahrenheit.
- 8.10 Calibration of Magnification - The computer-generated micrometer marker on the ADEM SEM will be checked annually against the NIST Reference Material 484. Tolerance is $\pm 10\%$. If out of tolerance it will be the Machine Custodian's responsibility to arrange for repair of the instrument so that it will be within tolerance.
- 8.11 Calibration of Gain - The gain on the ADEM EDS will be checked annually following the energy calibration instructions in the ADEM Operator's Manual and using Copper K and L alpha X-Ray lines. Tolerance is ± 20 eV. If the gain is found to be out of tolerance, it will be the Machine Custodian's responsibility to calibrate the gain as described in the Series II X-Ray Operator's Manuals.
- 8.12 Energy Resolution Check - The energy resolution of the ADEM EDS will be checked annually by measuring the full width at half maximum (FWHM) of the Manganese K alpha X-ray line which is the industry standard for detector resolution. This is a check for degradation of the detector ONLY: no adjustment or calibration will be made.
- 8.13 Lessons Learned
- During the performance of work, **ER Project participants** shall identify, document and submit lessons learned in accordance with QP-3.2, Lessons Learned, located at: http://erinternal.lanl.gov/home_links/Library_proc.htm.

9.0 REFERENCES

ER Project personnel may locate the ER Project Quality Management Plan at http://erinternal.lanl.gov/home_links/Library_proc.htm.

The following documents are cited within this procedure.

QP-2.2, Personnel Orientation and Training

QP-3.2, Lessons Learned

QP-4.2, Standard Operating Procedure Development

QP-4.3, Records Management

QP-5.7, Notebook Documentation for Environmental Restoration Technical Activities

ADEM Reference Manual (Tracor Northern; now Noran Instruments), Middleton, Wisconsin.

Instruction Manual, Ladd Vacuum Evaporator, cat. no. 3000, 27 pg., Burlington, Vermont.

Hummer II Operation and Maintenance Manual, Technics, 12 pgs. Alexandria, Virginia.

Jarosewich, E., J. A. Nelen, and J. A. Norberg, "Reference Samples for Electron Microprobe Analysis," in *GeoStandards Newsletter*, Vol. 4, No. 1, p. 43, April 1980.

10.0 RECORDS

The **users** of this SOP are responsible for submitting the following records (processed in accordance with QP-4.3) to the Records Processing Facility.

10.1 Notebook records of the sample handling and results of analysis relevant to production of scanning electron microscope data.

10.2 Data submittals of semi-quantitative SEM elemental analysis may be submitted to the ER electronic database; in general, such analyses will only be provided as part of the notebook record.

11.0 ATTACHMENTS

None

[Using a token card, click here to record "self-study" training to this procedure.](#)

If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.